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A. O. SMITH Corporation



MONTHLY PROGRESS REPORT
Contract Number NOw 62-0679-c (FBM)

EFFECT OF GLASS SURFACE CHEMISTRY
ON WETTING, BOND STRENGTH and BOND
LIFE OF AN EPOXY RESIN



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February 15, 1963

Gentlemen:

Progress Report #11 on Glass Surface Chemistry for Glass Fiber Reinforced Plastics for the period January 1 through January 31, 1963 is enclosed.

The test for measuring bond strength of the glass-resin coupling-agent system has been improved to the point where it is felt that reproducible results are being obtained. We are optimistic that further refinements will make the test even more sensitive.

Our contract time on this project has elapsed. However, there is a significant amount of money remaining. In view of the promising bond test results, it would appear that it would be worthwhile to continue the project at least until the original appropriation has been expended. A formal contract extension request has been submitted.

Very truly yours,

A. O. SMITH Corporation

A handwritten signature in cursive script, appearing to read 'F W Nelson'.

F W Nelson, Director
Ceramic Research and Development

FWN:mk

PROGRESS REPORT #11
on
GLASS SURFACE CHEMISTRY FOR GLASS FIBER REINFORCED PLASTICS
for the period
January 1, through January 31, 1963

I. Summary

A test for measuring the glass-resin bond strength in a dry environment appeared capable of distinguishing changes in the glass surface chemistry. The test indicated that the coupling agent A-1100 slightly improved the bond strength. It is felt that further refinements, such as the use of an invar instead of a teflon retainer, can make the test even more sensitive. Modifications of the test are presently being evaluated.

At the request of the program sponsor, an attempt will be made to correlate optically flat and as-cast glass surfaces with respect to bond life, bond strength, and wetting. With the cooperation of Owens-Corning, the bond life of HTS finished glass will be evaluated and compared with A-1100. These studies have been initiated.

Bond life studies in 100 per cent relative humidity showed this environment to be almost as severe as immersion in water.

II. Wetting Studies by the Captive Bubble Technique

Optical polishing changes the glass surface both physically and chemically. The significance of differences between the optically polished and virgin as-cast glass surface has not been established. Studies using a sample having an optically flat glass surface instead of the slightly curved as-cast surface would be simpler, particularly when measuring bond strength.

Previous wetting trials indicated that untreated, chemically cleaned, optically flat and as-cast E glass surfaces exhibited similar wetting behavior⁽¹⁾. Wetting trials will be made using the A-1100 treated optically flat glass surface

III. Bond Life Studies by the Flat Plate Test

An environment of 100 per cent relative humidity was compared with that of complete submersion in water. Epon 828 (CL) coatings on untreated, chemically cleaned E glass surfaces were placed in the air, instead of in the water, in the 190°F constant temperature bath. Debonding of the resin coatings resulted in 6 hours or less. Complete submersion in water resulted in debonding in 3 hours or less. The specimens having the A-1100 treated, chemically cleaned glass surfaces did not appear affected. Apparently, a 100 per cent relative humidity environment was about as severe as complete submersion in water.

Environments having lower water contents, such as room air, have not appeared damaging. Specimens having untreated, chemically cleaned glass surfaces placed in an oven (190°F air at 50% R. H.) exhibited no signs of debonding after 4 months. Untreated specimens on test in room temperature water failed in about 3 months or less.

These experiments show that a high moisture content of the environment is the chief cause of debonding. Temperature only accelerates the debonding by water.

Flat plate test specimens of A-1100 treated and untreated optically flat E glass surfaces were prepared and placed on test. With the cooperation of Owens-Corning, it is also planned to study the bond life of HTS finished E glass surfaces.

Table I summarizes the bond life studies.

IV. Bond Strength Studies by a Butt-Joint Chain Method

A test for measuring bond strength in a dry environment appeared capable of distinguishing changes in the glass surface chemistry. The bond strength specimens were prepared as previously shown⁽²⁾. However, the present method was different than previous column tests as follows:

1. Only bond failures (mirror interfaces) were considered.
2. The glass was tempered so that the glass surface, being stronger, did not fail
3. The teflon gasket design (Figure 1) was modified and the height of the resin column was reduced in order to decrease the tendency for resin leakage.
4. Placing a chain in the resin provided a self-aligning method for gripping the resin column.

Measurements by the butt-joint chain method have indicated that treatment with A-1100 slightly increased the bond strength. For 10 specimens having an untreated chemically cleaned glass surface, the mean bond strength was 990 psi, with a standard deviation of 210 psi. A total of 14 specimens were prepared, 4 of which did not fail at the bond. For 12 specimens having an A-1100 treated, chemically cleaned glass surface, the mean bond strength was 1320 psi, with a standard deviation of 190 psi. A total of 25 specimens were prepared, 13 of which did not fail at the bond. This data indicated that the chain method was a reproducible bond strength test. But it is felt that further refinements can make the test even more sensitive. Control of the bond area to an exact 1/8 inch diameter was difficult because the teflon deformed. The teflon gasket had to be re-machined to shape after each test. The use of invar as a retaining material is expected to eliminate the teflon deformation problem. However, the entire column designed as one continuous unit (Figure 1) could not be made from invar because machining the metal to a smooth surface was not practical. An invar retainer, similar to the teflon retainer, attached to an invar tube solved the machining problem, and is expected to improve the bond strength results. Evaluation of this refinement is presently in progress. At the request of the program sponsor, it is also planned to study optically flat E glass specimens. Sample preparation for this work is in progress.

V. References

1. Progress Report #4, Figures 4 and 5 (July 15, 1962).
2. Progress Report #9, Figures 1 and 2 (December 15, 1962).

TABLE I
BOND LIFE STUDIES BY FLAT PLATE TEST

Days to Failure Determined Visually

Type of Surface	Treatment	Days to Failure
Chemically clean	None	1
Chemically clean	1/2% A-1100	186
Contaminated	None	1
Contaminated	1/2% A-1100	186
Cleaved in Resin	None	1
Degassed	None	1
Degassed	1/2% A-1100	55
Degassed	10% A-1100	52*
Degassed	1/2% Z-6040	150*
Alkali-deficient (Prepared in air)	None	27*
Alkali-deficient	None	230*
Alkali-deficient	1/2% A-1100	147*
Alkali-rich	None	12
Alkali-rich	1/2% A-1100	49
Chemically clean, heated to 190°F before resin applied	None	1
As-cast (annealed)	None	1
Lightly sandblasted	None	7
Lightly sandblasted	1/2% A-1100	126*
Optically flat	None	1
Optically flat	1/2% A-1100	6*

*Still on test.

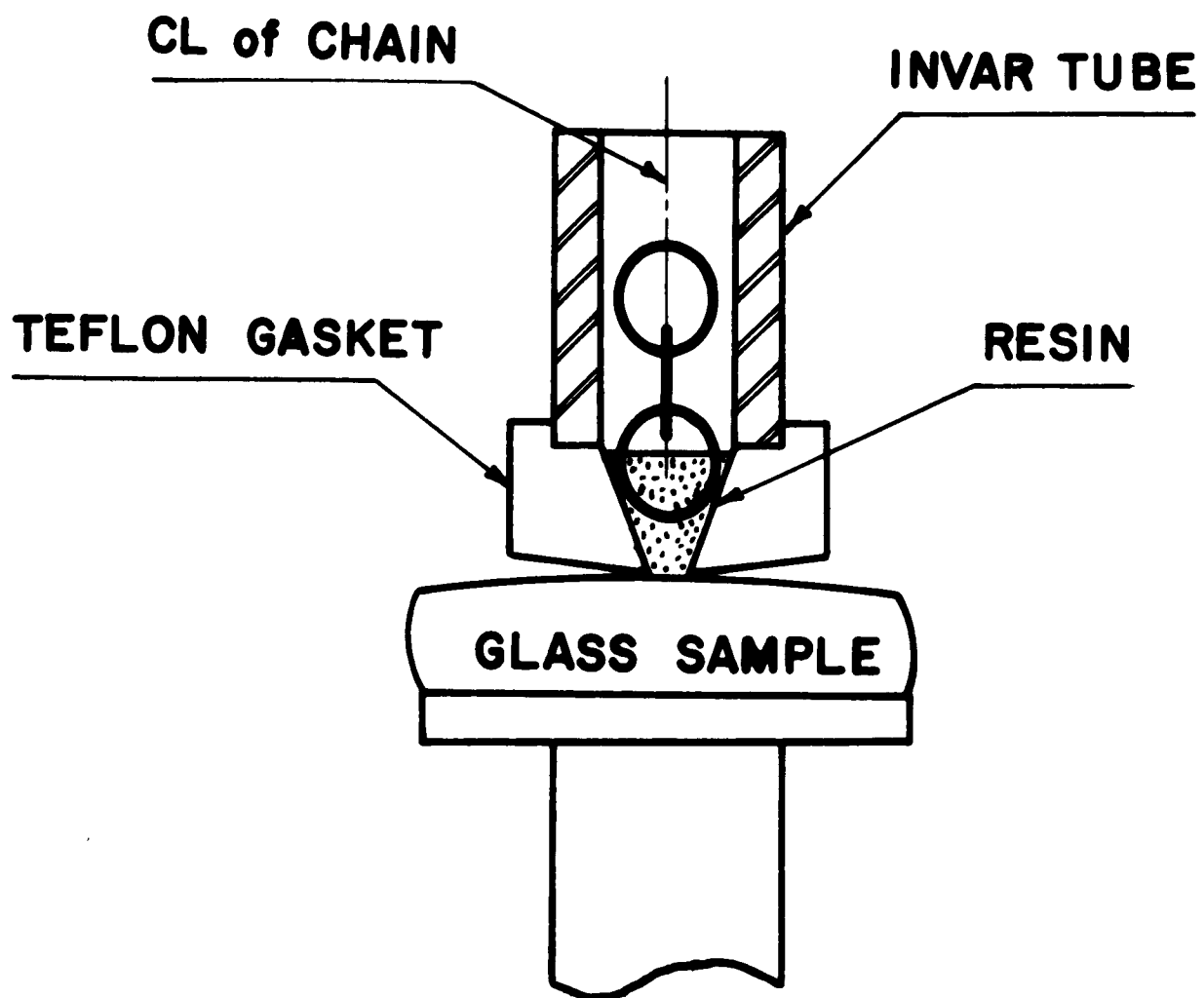


FIG.1 BOND STRENGTH BY CHAIN METHOD